SLOVENSKI STANDARD
SIST EN ISO 8988:1999

01-maj-1999

Polimerni materiali ï Fenolne smole ï Določevanje deleža heksametilentetramina ï Metodi po Kjeldahlu in s perklorno kislino (ISO 8988:1995)

Plastics - Phenolic resins - Determination of hexamethylenetetramine content - Kjeldahl method and perchloric acid method (ISO 8988:1995)

Kunststoffe - Phenolharze - Bestimmung des Hexamethylentetramingehaltes - Kjeldahl-Methode und Perchlorsäuremethode (ISO 8988:1995)

Plastiques - Résines phénoliques - Détermination de la teneur en hexaméthylenetétramine - Méthode Kjeldahl et méthode a l'acide perchlorique (ISO 8988:1995)

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Plastics - Phenolic resins - Determination of hexamethylenetetramine content - Kjeldahl method and perchloric acid method (ISO 8988:1995)

Plastiques - Résines phénoliques - Détermination de la teneur en hexaméthylènetétramine - Méthode Kjeldahl et méthode à l'acide perchlorique (ISO 8988:1995)

Kunststoffe - Phenolharze - Bestimmung des Hexamethylentetramingehaltes - Kjeldahl-Methode und Perchlorsäuremethode (ISO 8988:1995)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 1997, and conflicting national standards shall be withdrawn at the latest by August 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 8988:1995 has been approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 8988

Second edition
1995-05-01

Plastics — Phenolic resins — Determination of hexamethylenetetramine content — Kjeldahl method and perchloric acid method

Plastiques — Résines phénoliques — Détermination de la teneur en hexaméthylènetétramine — Méthode Kjeldahl et méthode à l'acide perchlorique

Reference number
ISO 8988:1995(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8988 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 8988:1989), of which it constitutes a minor revision.

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Plastics — Phenolic resins — Determination of hexamethylenetetramine content — Kjeldahl method and perchloric acid method

1 Scope

This International Standard specifies two methods for the determination of the hexamethylenetetramine ("hexa") content of phenolic resins. The two methods are equivalent. The Kjeldahl method described in clause 3 is not applicable if there are other components containing nitrogen in the phenolic resin. The perchloric acid method described in clause 4 is only applicable if there are no other basic or acidic additives in the resin.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

3 Kjeldahl method

WARNING — For safety reasons, the Kjeldahl determination must be carried out in a well ventilated hood.

3.1 General

This clause specifies a method for the determination of total nitrogen, expressed as hexamethylenetetramine, in phenolic resins.

The method is applicable to hexamethylenetetramine contents of $\geqslant 0,5$ % (m/m).

3.2 Principle

The hexamethylenetetramine in a test portion is converted to ammonium bisulfate by decomposition in hot concentrated sulfuric acid and in the presence of a catalytic mixture.

The ammonium bisulfate is converted to sodium sulfate and ammonia by reaction with sodium hydroxide.

The ammonia is distilled off and collected in hydrochloric acid.

The excess hydrochloric acid is titrated with a standard volumetric solution of sodium hydroxide in the presence of an indicator.

3.3 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, free of nitrogen, and only distilled water or water of equivalent purity.

3.3.1 Sulfuric acid, concentrated.

3.3.2 Kjeldahl catalytic mixture, consisting of 97 g of sodium sulfate decahydrate ($Na_2SO_4 \cdot 10H_2O$), 1,5 g of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) and 1,5 g of selenium (Se).

3.3.3 Sodium hydroxide, 30 % (m/m) solution.

3.3.4 Hydrochloric acid, $c(HCl) = 0,10$ mol/l.

3.3.5 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0{,}10$ mol/l.

3.3.6 Mixed indicator, solution.

Dissolve 60 mg of methyl red and 40 mg of methylene blue in 100 ml of ethanol.

3.4 Apparatus

Ordinary laboratory apparatus, plus the following:

3.4.1 Kjeldahl flask, capacity 250 ml or 300 ml, for the digestion.

3.4.2 Distillation apparatus (various models are available commercially).

3.4.3 Burette, capacity 50 ml, graduated in 0,1 ml steps, conforming with the requirements of ISO 385-1.

3.4.4 Analytical balance, accurate to 1 mg.

3.4.5 Silicon carbide grits, for use as anti-bumping granules.

3.5 Procedure

3.5.1 Digestion

Into a Kjeldahl flask (3.4.1), weigh 1 g to 2 g of phenolic resin to the nearest 1 mg. Add 5 g of the catalytic mixture (3.3.2) and 25 ml of concentrated sulfuric acid (3.3.1). Heat carefully until the colour of the mixture being digested changes from black or amber to clear. When clear, increase the rate of heating until 5 min beyond the time of the colour change and, possibly, boiling. Allow the digested liquid to cool almost to room temperature, just short of solidification. Add carefully 100 ml of water and transfer the solution quantitatively into the flask of the distillation apparatus, rinsing with water. Add a few silicon carbide grits (3.4.5) to prevent bumping. Add 30 % (m/m) NaOH solution (3.3.3) to this solution until an alkaline reaction is obtained. Then distill over the ammonia given off, together with water vapour, into a receiver containing 50 ml of hydrochloric acid (3.3.4). Continue the distillation until about 300 ml of water has been collected.

3.5.2 Titration

When the distillation is completed, add a few drops of mixed indicator solution (3.3.6) to the contents of the receiver and titrate the excess hydrochloric acid with sodium hydroxide solution (3.3.5), using the burette (3.4.3).

3.6 Expression of results

The hexamethylenetetramine content, expressed as a percentage by mass, is given by the formula

$$\frac{0{,}35(V_0 - V_1)}{m_0}$$

where

V_0 is the volume, in millilitres, of hydrochloric acid (3.3.4) in the receiver of the distillation apparatus;

V_1 is the volume, in millilitres, of sodium hydroxide solution (3.3.5) used in the back-titration;

m_0 is the mass, in grams, of the test portion.

3.7 Reproducibility

The results are reproducible to within 0,30 % (m/m) hexamethylenetetramine.

3.8 Number of determinations

Carry out the determination in duplicate. If the results differ by more than 5 %, repeat the determination, again in duplicate. If not, calculate the arithmetic mean of the two individual results.

4 Perchloric acid method

4.1 General

This clause specifies a method for the determination of hexamethylenetetramine in phenolic resins by direct titration. The results of the determination may be affected by the presence of acidic or basic additives. In such cases, the use of the Kjeldahl method is recommended.

The method is applicable to hexamethylenetetramine contents of $\geqslant 0{,}3$ %.

4.2 Principle

One of the tertiary amine groups of the hexamethylenetetramine in a test portion is determined by titration with perchloric acid.

4.3 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, free of nitrogen, and only distilled water or water of equivalent purity.

4.3.1 Hexamethylenetetramine, dry.

4.3.2 Acetone.

4.3.3 Perchloric acid, 70 % (V/V) solution.

WARNING — Perchloric acid is dangerous in the presence of organic matter since an explosion can occur if the perchloric acid is in excess.

4.4 Apparatus

Ordinary laboratory apparatus, plus the following:

4.4.1 Magnetic stirrer.

4.4.2 Automatic burette, nominal volume at least 15 ml, graduated in 0,1 ml steps, with a stopcock made of polytetrafluoroethylene.

4.4.3 Beakers, capacity 100 ml.

4.4.4 Graduated cylinder, capacity 1 000 ml.

4.4.5 Analytical balances, accurate to 0,1 mg and 1 mg, respectively.

4.4.6 pH-meter.

4.5 Procedure

4.5.1 Preparation and titration of solution of perchloric acid in acetone

Introduce 8 ml of perchloric acid solution (4.3.3) into the 1 000 ml graduated cylinder (4.4.4), and dilute to 1 000 ml with acetone (4.3.2).

Standardize the resulting solution with hexamethylenetetramine (4.3.1) as described below.

Weigh, to the nearest 0,1 mg, about 150 mg to 170 mg of hexamethylenetetramine (4.3.1) into a 100 ml beaker (4.4.3).

Add 30 ml to 40 ml of acetone (4.3.2), and titrate as described (4.5.2).

NOTE 1 Darkening of the solution will not affect the titration results.

The titre T, expressed in milligrams of hexamethylenetetramine per millilitre of solution, is given by the formula

$$\frac{m_1}{V_2}$$

where

m_1 is the mass, in milligrams, of hexamethylenetetramine;

V_2 is the volume, in millilitres, of perchloric acid solution needed to reduce the pH to just below zero.

4.5.2 Titration

Into a 100 ml beaker weigh, to the nearest 1 mg, a quantity of phenolic resin equal to 100 times the titre determined as in 4.5.1, add 30 ml to 40 ml of acetone (4.3.2) and insert a magnetic stirring rod. Place the beaker on the magnetic stirrer (4.4.1). Insert the glass electrode of the pH-meter (4.4.6) and switch on the stirrer and the pH-meter. When the resin has dissolved, add the perchloric acid solution, prepared as in 4.5.1, slowly dropwise until the pH-value drops suddenly below zero. As the resin dissolves in acetone more rapidly than hexamethylenetetramine, the pH-reading may increase to above zero because residual hexamethylenetetramine may still be in the process of dissolving. Continue the titration until the pH-reading remains constant, slightly below zero.

4.6 Expression of results

The hexamethylenetetramine content, expressed as a percentage by mass, is given by the formula

$$\frac{V_2\, T \times 100}{m_0}$$

where

V_2 is the volume, in millilitres, of perchloric acid solution used for the titration;

T is the titre, expressed in milligrams of hexamethylenetetramine per millilitre, of the perchloric acid solution, as determined in 4.5.1;

m_0 is the mass, in milligrams, of the test portion.

4.7 Number of determinations

Carry out the determination in duplicate. If the results differ by more than 5 %, repeat the determination,